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Indian Standard

SPECIFICATION FOR FERROUS SULPHATE, HEPTAHYDRATE

(Second Revision)

First Reprint JUNE 1994

UDC 661.872.532-12

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 5 *April* 1983

Indian Standard
SPECIFICATION FOR FERROUS SULPHATE, HEPTAHYDRATE
(Second Revision)

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Indian Standard

SPECIFICATION FOR FERROUS SULPHATE, HEPTAHYDRATE

(Second Revision)

0. FOREWORD

0.1 This Indian Standard (Second Revision) was adopted by the Indian Standards Institution on 30 June 1982, after the draft finalized by the Inorganic Chemicals (Miscellaneous) Sectional Committee had been approved by the Chemical Division Council.

0.2 This standard was originally issued in 1950. The Inks and Allied Products Sectional Committee suggested that the requirements for ferrous sulphate used in the ink industry may also be covered under this standard. Considering this and the progress made by the industry since the publication of this standard, the Sectional Committee, responsible for the preparation of this standard decided to revise it for including a pure grade suitable for inks and pharmaceutical industries and purification of water, and an analytical reagent grade. Modifications were also made in the method of sampling and some of the methods of test.

0.3 The second revision of this standard has been undertaken to exclude the pure grade which included the pharmaceutical grade, as the same is already covered by the Indian Pharmacepoeia. The percentage purity of technical grade has also been raised to minimum 97 percent, for meeting the requirements for purification of water and ink industry.

0.4 This material is widely used, in various industries, such as, fertilizer, as food and feed supplement, as wood preservative, as weed killer, as dye for leather, in prevention of chlorosis of plants, in process engraving and lithography, in etching aluminium, in writing inks, in water treatment, in qualitative analysis and chemical testing, in chemical processes, and for making other iron salts like oxides.

0.5 This standard contains clause 4.1 which calls for agreement between the purchaser and the supplier.

0.6 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for ferrous sulphate, heptahydrate.

2. GRADES

2.1 There shall be two grades of the material as follows:

a) Technical Grade (TECH)	For textile industries, for use in the production of iron oxide pigments for paints, for ink industry and for purification of water.
b) Analytical Reagent Grade (AR)	For use as a reagent.

3. REQUIREMENTS

3.1 Description — The material shall be in the form of green or bluish green crystals free from visible impurities.

3.2 Solubility

3.2.1 Ten grams of the material of technical grade when dissolved in 100 ml of freshly boiled and cooled water at 27 ± 2°C shall give a pale yellowish green solution which shall be not more than faintly turbid.

3.2.2 Analytical reagent grade of the material shall be soluble in 0.5 part of boiling water producing a clear green coloured solution. It shall be practically insoluble in alcohol.

3.3 The material shall also comply with the requirements given in Table 1, when tested according to the methods prescribed in Appendix A. Reference to the relevant clauses of Appendix A is given in col 5 of the table.

3.3.1 *Additional Requirements for Material Required for Purification of Water* — The technical grade of the material when required for purification of water shall also comply with the requirements given in Table 2 in addition to those given under col 3 of Table 1.

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*Rules for rounding off numerical values (*revised*).

TABLE 1 REQUIREMENTS FOR FERROUS SULPHATE, HEPTAHYDRATE

(*Clause* 3.3)

SL No.	CHARACTERISTIC	REQUIREMENT FOR GRADE		METHOD OF TEST (REF TO CL NO. IN APPENDIX A)
		Tech	Ar	
(1)	(2)	(3)	(4)	(5)
i)	Ferrous sulphate, heptahydrate ($FeSO_4 . 7H_2O$), percent by mass	97.0 (*Min*)	99.0 to 101.0	A-3
ii)	Free acid (as H_2SO_4), percent by mass, *Max*	0.2	—	A-4
iii)	*p*H, not less than	—	3.7	A-5
iv)	Iron (as Fe), percent by mass, *Max*	0.2	0.025	A-6
v)	Manganese (as Mn), percent by mass, *Max*	—	0.05	A-7
vi)	Chlorides (as Cl), percent by mass, *Max*	—	0.0005	A-8
vii)	Phosphates (as PO_4), percent by mass, *Max*	—	0.001	A-9
viii)	Heavy metals (as Pb), parts per million, *Max*	—	25	A-10
ix)	Arsenic (as As_2O_3), parts per million, *Max*	—	1	A-11
x)	Copper	—	To pass test	A-12
xi)	Zinc	—	To pass test	A-13
xii)	Alkaline salts and alkaline earths, percent by mass, *Max*	—	0.15	A-14

TABLE 2 ADDITIONAL REQUIREMENTS FOR FERROUS SULPHATE, HEPTAHYDRATE FOR PURIFICATION OF WATER

(*Clause* 3.3.2)

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST (REF TO CL NO. IN APPENDIX A)
(1)	(2)	(3)	(4)
i)	Heavy metals (as Pb), parts per million, *Max*	50	A-10
ii)	Arsenic (as As_2O_3), parts per million, *Max*	8	A-11

4. PACKING AND MARKING

4.1 Packing — The material shall be packed in jute or canvas bags with a polyethylene liner, or as agreed to between the purchaser and the supplier. Technical grade of the material shall be packed in jute bags with polyethylene liner securely sealed so as to protect it from outside air and moisture.

Analytical reagent grade shall be packed in glass or polyethylene bottles and properly sealed to avoid contact with air.

4.2 Marking — The containers shall be securely closed and legibly marked with the following information:

a) Name and grade of the material;
b) Manufacturer's name and recognized trade-mark, if any;
c) Mass of the material in the container;
d) Month and year of manufacture; and
e) Lot number to enable the batch of manufacture to be traced from records.

4.2.1 In case of AR grade of the material, data of analysis of the material as laid down in col 4 of Table 1 shall also appear on the label.

4.2.2 The containers may also be marked with the Standard Mark.

4.2.3 The use of the Standard Mark is governed by the provisions of Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

5. SAMPLING

5.1 The procedure for taking out representative samples from a lot, the number of tests and the criteria for conformity of the material to the requirements of the specification shall be as prescribed in Appendix B.

APPENDIX A

(*Clauses* 3.3 and 3.3.1)

METHODS OF TEST FOR FERROUS SULPHATE, HEPTAHYDRATE

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be used in tests.

NOTE — 'Pure Chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

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*Specification for water for general laboratory use (*second revision*).

A-2. PREPARATION OF SAMPLE

A-2.1 Procedure — Powder 200 g of the material to pass through 1.00 mm IS Sieve [*see* IS : 460 (Part I)-1978*]. Keep the prepared sample in a clean glass-stoppered bottle for test purposes.

A-3. DETERMINATION OF FERROUS SULPHATE, HEPTAHYDRATE (AS $FeSO_4$, H_2O)

A-3.0 Outline of the Method — Ferrous sulphate content is determined by titration with standard potassium permanganate solution.

A-3.1 Reagents

A-3.1.1 *Dilute Sulphuric Acid* — 4 N approximately.

A-3.1.2 *Standard Potassium Permanganate Solution* — 0.1 N (*see* 51 of IS : 2316-1968†).

A-3.1.3 *Ortho-Phosphoric Acid*

A-3.2 Procedure — Weigh accurately about 5 g of the prepared sample (*see* A-2.1) and dissolve it in water. Add 1 to 2 ml of dilute sulphuric acid and make up the solution to 250 ml in a volumetric flask. Pipette out exactly 50 ml of the solution in a flask. Add 10 ml of dilute sulphuric acid and 2 ml of ortho-phosphoric acid and titrate with standard potassium permanganate solution.

A-3.3 Calculation

Iron sulphate, heptahydrate (as $FeSO_4$, $7H_2O$), percent by mass $= \dfrac{139 \times V \times N}{M}$

where

V = volume of standard potassium permanganate solution used,
N = normality of standard potassium permanganate solution, and
M = mass in g of the material taken for the test.

A-4. TEST FOR FREE ACID AS SULPHURIC ACID (AS H_2SO_4)

A-4.1 Reagents

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*Specification for test sieves: Part I wire cloth tests sieves (*second revision*).

†Methods of preparation of standard solutions for colorimetric and volumetric analysis (*first revision*).

A-4.1.1 *Methyl Orange Indicator* — Dissolve 0.01 g of methyl orange in 100 ml of water.

A-4.1.2 *Standard Sodium Hydroxide Solution* — 0.1 N.

A-4.2 Procedure

A-4.2.1 Weigh accurately about 4 g of the prepared sample (*see* A-2.1) and dissolve it in 100 ml of water. Add 4 drops of methyl orange indicator. Divide it into two equal portions. Add 0.8 ml of standard sodium hydroxide solution to one of the portions keeping the other portion for comparison of the colour. For pure grade use 8 g of the prepared sample (A-2.1).

A-4.2.2 The limit prescribed in Table 1 shall be taken as not having been exceeded if the purplish colour of the material under test disappears on the addition of standard sodium hydroxide solution.

A-5. DETERMINATION OF *p*H

A-5.1 Procedure — Dissolve 5.0 g of the prepared sample (A-2.1) in 100 ml of freshly boiled and cooled water and determine the *p*H by means of a *p*H meter using glass electrodes.

A-6. DETERMINATION OF IRON (As Fe)

A-6.0 General — Ferric iron is found by comparing the colour produced with ammonium thiocyanate solution against the colour of standard iron solution.

A-6.1 For Technical Grade

A-6.1.1 *Reagents*

A-6.1.1.1 *Concentrated hydrochloric acid* — *See* IS : 265-1976*.

A-6.1.1.2 *Dilute sulphuric acid* — 1:1(*v*/*v*).

A-6.1.1.3 *Ammonium thiocyanate solution* — 10 percent (*m*/*v*).

A-6.1.1.4 *Standard iron solution* — Dissolve 0.702 g of ammonium ferrous sulphate [$FeSO_4.(NH_4)_2SO_4.6H_2O$] in water containing 10 ml of dilute sulphuric acid and dilute with water to 1 000 ml. Dilute 10 ml of the solution to 100 ml. One millilitre of the dilute solution contains 0.01 mg of iron (as Fe).

A-6.1.2 *Apparatus*

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*Specification for hydrochloric acid (*second revision*).

A-6.1.2.1 *Nessler cylinders* — 50 ml capacity.

A-6.1.3 *Procedure* — Dissolve 2.5 g of the prepared sample (*see* **A-2.1**) in water containing 2.5 to 3 ml of sulphuric acid. The solution is made up to 250 ml and 5 ml of the solution is taken in the Nessler cylinder. Add 2 ml of hydrochloric acid and 5 ml of ammonium thiocyanate solution and the volume is made up to 50 ml with water.

Carry out a control test using 10 ml of standard iron solution in the other cylinder.

A-6.1.3.1 The limit prescribed in Table 1 shall be taken as not having been exceeded, if the intensity of the colour produced with the material is not greater than that produced in the control test.

A-6.2 For AR Grade

A-6.2.1 *Procedure* — Follow the same procedure as in **A-6.1.2**, but take 5 g sample and make up the volume to 250 ml. Take 10 ml of this solution in a Nessler cylinder. The colour developed should not be darker than that developed by 5 ml of standard iron solution.

A-7. DETERMINATION OF MANGANESE (AS Mn)

A-7.0 Outline of the Method — Manganese is determined by comparing the colour produced with sodium bismuthate against the colour of standard potassium permanganate solution.

A-7.1 Apparatus

A-7.1.1 *Nessler Cylinders* — 50 ml capacity.

A-7.2 Reagents

A-7.2.1 *Concentrated Nitric Acid* — *See* IS : 264-1976*.

A-7.2.2 *Sodium Bismuthate* — powder.

A-7.2.3 *Standard Potassium Permanganate Solution* — Prepare a dilute solution of potassium permanganate by diluting to 50 ml in a volumetric flask, a quantity of a recently standardized solution in accordance with the following formula:

$$A = \frac{0.455\ 1}{N}$$

---

*Specification for nitric acid (*second revision*).

where

A = volume in ml of the standard potassium permanganate solution to be made up to a volume of 50 ml, and

N = normality of the standard potassium permanganate solution.

NOTE 1 — One millilitre of this solution is equivalent to 0.1 mg of manganese (Mn).

NOTE 2 — The solution shall be prepared afresh.

A-7.3 Procedure — Dissolve 1 g of the prepared sample (*see* **A-2.1**) in 40 ml of water and 10 ml of concentrated nitric acid. Boil off nitrous fumes. Add 1 g of sodium bismuthate and about 1 ml of phosphoric acid. Shake vigorously for five minutes, and make up the volume with water to 50 ml. Carry out a control test using 3 ml of standard potassium permanganate solution in the other Nessler cylinder.

A-7.3.1 The limit prescribed in Table 1 shall be taken as not having been exceeded, if the intensity of the colour produced with the material is not greater than that produced in the control test.

A-8. DETERMINATION OF CHLORIDES (AS Cl)

A-8.1 Reagents

A-8.1.1 *Dilute Nitric Acid* — 5 N approximately.

A-8.1.2 *Hydrogen Peroxide* — 30 volumes.

A-8.1.3 *Silver Nitrate Solution* — 5 percent (*m/v*).

A-8.2 Procedure — Dissolve 2.0 g of the prepared sample (*see* **A-2.1**) in 45 ml of water, add 2 ml of dilute nitric acid, 3 ml of hydrogen peroxide and 1 ml of silver nitrate solution. The limit prescribed in Table 1 shall be taken as not having been exceeded if no opalescence is produced.

A-9. DETERMINATION OF PHOSPHATES (AS PO_4)

A-9.1 Apparatus

A-9.1.1 *Separating Funnels* — 250 ml capacity.

A-9.1.2 *pH Meter* — with glass and calomel electrodes.

A-9.2 Reagents

A-9.2.1 *Dilute Sulphuric Acid* — approximately 5 N.

A-9.2.2 *Ammonium Molybdate*

A-9.2.3 *Concentrated Hydrochloric Acid* — *See* IS : 265-1976*.

A-9.2.4 *Dilute Hydrochloric Acid* — approximately 5 N.

A-9.2.5 *Ethyl Ether*

A-9.2.6 *Stannous Chloride Solution* — Dissolve 2 g of stannous chloride ($SnCl_2.2H_2O$) in concentrated hydrochloric acid and dilute to 100 ml with water.

A-9.2.7 *Standard Phosphate — Silicate Solution* — Dissolve 1.43 g of potassium dihydrogen phosphate (KH_2PO_4) and 2.365 g of sodium silicate ($Na_2SiO_3.9H_2O$) in water and make up to 1 000 ml. Store this solution in a waxed bottle. Dilute 1 ml of this solution to 100 ml with water immediately before use. One millilitire of this diluted solution is equivalent to 0.01 mg of phosphate (as PO_4) and 0.005 mg of silica (as SiO_2).

A-9.3 Procedure — Weigh accurately 1.000 g of the prepared sample (*see* **A-2.1**) and dissolve in 50 ml of water in a platinum dish. Digest on a steam bath for 20 minutes. Cool, neutralize with dilute sulphuric acid to a *p*H of about 4, and dilute to about 75 ml. Take 1 ml of standard phosphate-silicate solution and make up to about 75 ml. Add 0.5 g of ammonium molybdate to each solution and when it is dissolved, adjust the *p*H to about 2 by adding dilute hydrochloric acid. Check the *p*H with the *p*H meter. Heat both the solution to boiling, cool to room temperature, add 10 ml of concentrated hydrochloric acid to each, and dilute to 100 ml with water. Transfer the solutions to separating funnels, add 35 ml of ether to each, shake vigorously and allow to separate. Wash the ether phase of each by shaking with 10 ml of dilute hydrochloric acid, allow to separate, and draw off; and discard this aqueous phase. Add 0.2 ml of freshly prepared stannous chloride solution to each ether extract and shake. If the ether extracts are turbid, wash with 10 ml of dilute hydrochloric acid.

A-9.4 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of the blue colour produced in the test with the material is not greater than that produced in the control test.

A-10. DETERMINATION OF LEAD (AS Pb)

A-10.0 Outline of the Method — Lead is determined colorimetrically in Nessler cylinders using saturated hydrogen sulphide solution.

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*Specification for hydrochloric acid (*second revision*).

A-10.1 Apparatus

A-10.1.1 *Nessler cylinders* — 50 ml capacity.

A-10.2 Reagents

A-10.2.1 *Concentrated Hydrochloric Acid* — *See* IS : 265-1976*.

A-10.2.2 *Standard Lead Solution* — Dissolve 1.60 g of lead nitrate i water and make up the volume to 1 000 ml. Transfer exactly 10 ml of th solution into a volumetric flask and dilute it again with water to 1 000 m mark. One millilitre of this solution contains 0.01 mg of lead (as Pb). The dilute solution shall be freshly prepared.

A-10.2.3 *Dilute Acetic Acid* — approximately 1 N.

A-10.2.4 *Hydrogen Sulphide Solution* — saturated.

A-10.3 Procedure — Weigh accurately 1.0 g of the prepared sample (*see* A-2.1) and add 15 ml of water. Add 6 ml of concentrated hydrochloric acid and evaporate to dryness on a water bath. Dissolve the residue in water and transfer this solution into a Nessler cylinder. Dilute it with water to 30 ml and add 1 ml of acetic acid and 10 ml of hydrogen sulphide solution. In a second Nessler cylinder, carry out a control test using 5 ml of standard lead solution in case of material required for purification of water and 2.5 ml in case of analytical reagent grade, and the same quantities of other reagents in the same total volume of the mixture.

A-10.3.1 Dilute the contents of each cylinder to 50 ml and shake well. Compare the colour produced. The limits prescribed in Tables 1 and 2 shall be taken as not having been exceeded if the intensity of colour produced with the material is not greater than that produced in the control test.

A-11. DETERMINATION OF ARSENIC (AS As_2O_3)

A-11.1 Procedure — Dissolve 1.0 g of the material in minimum amount of dilute sulphuric acid (approximately 4 N). Carry out the test for arsenic as prescribed in IS : 2088-1971†, using for comparison a strain obtained with the following quantities of arsenic trioxide (as As_2O_3):

a) 0.009 mg in case of material required for purification of water, and

b) 0.001 mg in case of analytical reagent grade.

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*Specification for hydrochloric acid (*second revision*).

†Methods for determination of arsenic (*first revision*).

A-12. TEST FOR COPPER

A-12.0 Principle — Copper is tested by the appearance of blue colour with ammonium hydroxide solution.

A-12.1 Reagent

A-12.1.1 *Dilute Ammonium Hydroxide Solution* — approximately 10 percent (*m/v*).

A-12.2 Procedure — Dissolve 1.0 g of the prepared sample (*see* **A-2.1**) in 100 ml of water and add few drops of concentrated nitric acid and warm the solution for 1 or 2 minutes, add to it gradually dilute ammonium hydroxide solution until the solution is alkaline to litmus and then add few drops more. Boil the solution and then filter. Note the colour of the filtrate.

A-12.2.1 The material shall be taken to have satisfied the requirements of the test if no blue colour is produced.

NOTE — Carbon tetrachloride and isoamyl alcohol may be used for extracting and thereby to intensify the colour and/or to eliminate turbidity. The solvents are added after the colour has developed. The mixtures are then shaken to cause the colour to pass into the organic layer. Care shall be taken that the solvents themselves do not contain traces of metals. It is desirable to acidify the aqueous solution immediately prior to the extraction. which is slow, from an alkaline solution.

A-13. TEST FOR ZINC

A-13.1 Reagents

A-13.1.1 *Concentrated Nitric Acid* — *See* IS : 264-1976*.

A-13.1.2 *Concentrated Ammonium Hydroxide* — 20 percent NH_3 (*m/m*).

A-13.1.3 *Dilute Hydrochloric Acid* — 5 N.

A-13.1.4 *Potassium Ferrocyanide Solution* — 5 percent (*m/v*).

A-13.2 Procedure — Weight 2.0 g of the prepared sample (**A-2.1**) and dissolve in 30 ml of water, add 3 ml of concentrated nitric acid and boil to expel nitrous fumes. Add 20 ml of water and 10 ml of ammonium hydroxide and filter. Neutralize the filtrate with dilute hydrochloric acid and add 1 ml of the acid in excess and then add 2 ml of potassium ferrocyanide solution.

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*Specification for nitric acid (*second revision*).

A-13.2.1 The material shall pass the requirement of the test if no turbidity appears in 10 minutes.

A-14. DETERMINATION OF ALKALINE SALTS AND ALKALINE EARTHS

A-14.1 Reagents

A-14.1.1 *Concentrated Nitric Acid* — *See* IS : 264-1976*.

A-14.1.2 *Dilute Ammonium Hydroxide* — 15 percent NH_3 (*m/m*).

A-14.2 Procedure — Weigh 1.0 g of the prepared sample (*see* **A-2.1**) and dissolve in 10 ml of water. Oxidize by warming with a few drops of concentrated nitric acid and make alkaline with dilute ammonium hydroxide. Filter and wash several times with a minimum quantity of hot water until the filtrate is neutral to litmus. Collect the filtrate in a suitable porcelain or silica crucible and evaporate to dryness. Ignite the residue, cool and weigh to constant mass.

A-14.3 Calculation

$$\text{Alkaline salts and alkaline earths, percent by mass} = \frac{M_1}{M_2} \times 100$$

where

M_1 = mass in g of the residue after ignition, and
M_2 = mass in g of the material taken for the test.

APPENDIX B

(*Clause* 5.1)

SAMPLING OF FERROUS SULPHATE, HEPTAHYDRATE

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

B-1.1 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

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*Specification for nitric acid (*second revision*).

B-1.2 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

B-1.3 The samples shall be placed in clean, dry and air-tight or any other suitable containers on which the material has no action.

B-1.4 Each sample container shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling and the year of manufacture of the material.

B-2. SCALE OF SAMPLING

B-2.1 Lot — All the containers in a single consignment of the material of one grade and drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of different grades or batches of manufacture, the containers belonging to the same grade and batch shall be grouped together and each such group shall constitute a separate lot.

B-2.1.1 Samples shall be tested from each lot for ascertaining conformity of the material to the requirements of the specification.

B-2.2 The number (n) of containers to be chosen from a lot shall depend on the size of the lot (N) and shall be in accordance with col. 1 and 2 of Table 3.

B-2.3 The containers to be selected for sampling shall be chosen at random from the lot and for this purpose random number tables shall be used. In case such tables are not available, the following procedure may be adopted:

> Starting from any container, count them 1, 2, 3........up to r and so on in a systematic manner, where r is the integral part of N/n. Every rth container thus counted shall be taken out for drawing supplies.

TABLE 3 NUMBER OF CONTAINERS TO BE SELECTED

(*Clause* B-2.2)

Lot Size N (1)	Number Of Containers To be Selected n (2)
3 to 50	3
51 to 200	4
201 to 400	5
401 to 650	6
651 and above	7

B-3. TEST SAMPLES AND REFEREE SAMPLE

B-3.1 Preparation of Test Samples

B-3.1.1 Draw with an appropriate sampling instrument a small portion of the material from different parts of each container selected. The total quantity of the material drawn from each container shall be sufficient to conduct the test for all the characteristics given under 3 and shall not exceed 1 kg.

B-3.1.2 Thoroughly mix all portions of the material drawn from the same container. Out of these portions a small but equal quantity shall be taken from each selected container and shall be well mixed up together so as to form a composite sample weighing not less than 600 g. This composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third to be used as referee sample.

B-3.1.3 The remaining portion of the material from each container (after a small quantity needed for the formation of composite sample has been taken) shall be divided into three equal parts, each part weighing not less than 100 g. These parts shall be immediately transferred to thoroughly dried bottles which are then sealed air-tight with stoppers and labelled with all the particulars of sampling given under **B-1.4**. The material in each such sealed bottle shall constitute an individual test sample. These individual samples shall be separated into three identical sets of samples in such a way that each set has an individual test sample representing each container selected. One of these three sets shall be sent to the purchaser, another to the supplier and the third shall be used as referee sample.

B-3.2 Referee Sample — The referee sample shall consist of the composite sample (*see* **B-3.1.2**) and a set of individual samples (*see* **B-3.1.3**) marked for this purpose and shall bear the seals of the purchaser and the supplier. These shall be kept at a place agreed to between the purchaser and the supplier and shall be used in case of dispute between the two.

B-4. NUMBER OF TESTS

B-4.1 Tests for the determination of ferrous sulphate, heptahydrate shall be conducted on each of the individual samples for all the grades.

B-4.2 Tests for the remaining characteristics shall be conducted on the composite sample.

B-5. CRITERIA FOR CONFORMITY

B-5.1 For Individual Samples

B-5.1.1 *For Ferrous Sulphate* — The test results for ferrous sulphate, heptahydrate shall be recorded, and the mean and the range for these test results shall be calculated as follows:

Mean ($\bar{X}$) = Sum of the test results divided by the number of test results, and
Range (R) = The difference between the maximum and the minimum values of the test results.

The value of expression ($\bar{X} \pm 0.6R$) shall be calculated. If the values of this expression lie within the limits specified for the relevant grade in Table 1, the lot shall be declared to have satisfied the requirement for this characteristic.

B-5.2 For Composite Sample — The test results on the composite sample shall meet the corresponding requirements specified in Table 1 for the relevant grade.

A-5.3 A lot shall be declared as conforming to the specification if it satisfies the requirements for each of the characteristics listed in Table 1 for the relevant grade.

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